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(56) Documents Cited

US 4442253 A US 4421882 A  
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JP610231051

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(54) Abstract Title

Thermoplastic elastomers

(57) This invention relates to high performance thermoplastic elastomers (TPEs) in different colours with good weather performance and compression set, and use of the same.

The thermoplastic elastomeric composition comprises PVC, homo- or copolymer, EPDM (ethylene propylene diene monomer) and/or EPR and/or other polyolefinic elastomers, polycaprolactone (PCL) and/or in situ polymerised  $\epsilon$ -caprolactone, and conventionally known additives for preparation of PVC.

Possible product areas include, but are not restricted to uPVC window seals, vehicle window seals and waistrails, technical and appliance hose, automotive components and speciality electrical cabling, both appliance and data.

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### THERMOPLASTIC ELASTOMERS

This invention relates to high performance thermoplastic elastomers (TPEs) in different colours with good weather performance and compression set, and use of the same.

Window seals involve two main categories, glazing gaskets and weather seals. The former class is designed to remain in a fixed position, whereas weather seals undergo frequent compression and recovery cycles in service. Higher performance elastomeric materials are necessary to meet these service criteria over a range of ambient temperatures. Flexible PVC (polyvinyl chloride) has limited application as a thermoplastic elastomer due to low heat distortion temperatures and poor permanent compression strain properties. Vulcanized elastomers (thermosets) overcome those limitations, but are difficult to process on thermoplastic equipment, usually difficult to flame retard, not recyclable and often have poor oil, chemical and ozone resistance. In thermoset rubbers reinforcement with high loadings of carbon black is necessary to achieve appropriate mechanical performance, resulting in black products.

PVC is miscible with a number of polymers due to its polarity, and has a long history of blending. Blends of PVC with i) acrylic impact modifier or ii) NBR (nitrile butadiene rubber) appeared in 1936 as the first commercial thermoplastic blends. Still today most PVC-based TPEs are blends of PVC and plain or crosslinked (thermoplastic vulcanizates, TPVs) NBR, made for e.g. automotive applications, oil-resistant hoses, weather seals and cable insulation. A great disadvantage of PVC-NBR blends is the unsaturated backbone of NBR, which provides poor ozone stability. Like many other TPEs it shows poor colour hold in light colours and hence black colouration is required to overcome this effect. TPEs in white or other light colours are of interest, particularly in gaskets and seals for white uPVC (unplasticized polyvinyl chloride) windows where colour hold must be as good as that required for the frame. Thus, it is highly desired to be able to provide high quality TPEs with good weather performance in any colour.

It was surprisingly found by the inventors that by replacing NBR with EPDM (ethylene propylene diene monomer), EPR (ethylene propylene rubber) or other polyolefinic elastomers high quality TPEs with good weather performance in any colour were produced. EPDM is resistant towards heat, ozone and oxygen, and is able to tolerate high concentration of filler and oil, yet retaining good physical properties.

There are previously known several descriptions of rubber-toughened plastics (4-10 wt % e.g. EPDM) with improved impact strength at high and low temperatures. These blends are however different from our TPEs, which have a higher EPDM content. One reference describes blends of PVC (0-100 parts per hundred (phr)) and EPDM with improved tensile properties, employing NBR-18 as compatibilizer (EPDM:NBR 90:10). The tensile properties are far below the achievements of the present invention. In the patent literature blends of PVC or PVC copolymers with crosslinked NBR (JP61195144, EP353525, GB2271115), crosslinked carboxylated NBR (US4826910) and mixtures of crosslinked NBR (97-70 wt %) and EPDM (3-30 wt %) (JP07053820, JP07053819) are described. All blends have low compression set and good tensile properties, but have the aforementioned drawbacks of NBR.

PVC and EPDM, EPR or most other polyolefinic elastomers are completely incompatible. To get a satisfactory blend a compatibilizer is needed. In US.Pat.No. 4,442,253 and 4,421,882 blends of sulphonated EPDM neutralized with amine-terminated polycaprolactone (PCL) and e.g. PVC are described. Sulphonated EPDM is more expensive than unmodified EPDM and is therefore less attractive commercially. The elongation at break is also poor (10-50%) for these blends.

It is an object of the present invention to provide a new "dynamic" TPE which combines coextrudability and comouldability to rigid PVC and high performance elastomeric recovery over a wide service temperature range with good colour hold in light colours. This object is achieved by providing a TPE consisting of PVC, EPDM, EPR and/or other polyolefinic elastomers and PCL.

Possible product areas include, but are not restricted to uPVC window seals, vehicle window seals and waistrails, technical and appliance hose, automotive components and speciality electrical cabling, both appliance and data.

The present invention provides a thermoplastic elastomeric composition comprising

- (i) from 10-70 wt % PVC, plain or as a copolymer,
- (ii) from 15-70 wt % EPDM or EPR, plain or grafted,
- (iii) from 5-50 wt % polycaprolactone (PCL) ,
- (iv) from 0-10 wt % transesterification catalyst ,
- (v) from 0 to 10 wt % sulphur-, peroxy curing or chemical crosslinking agent,
- (vi) from 0 to 30 wt % coagent for the curing agent,
- (vii) from 0-50 wt % plasticizer and
- (viii) from 0.3-6 wt % stabilizers and processing aids.

The thermoplastic vulcanizates produced according to the present invention exhibit good tensile properties, compression set and thermal stability provided that appropriate processing conditions are used.

The PVC used in the present invention may be any resin known, including but not restricted to graft- and copolymers such as VC (vinyl chloride)/VA (vinyl acetate), VC/VDC (vinylidene chloride), VC/AN (acrylonitrile), VC/P (propylene), VC/E (ethylene), VC/EVA (ethylene/vinyl acetate), VC/TFCE(trifluorochloroethylene), VC/hydroxy-/ethyl-/propylmethacrylate, VC/fumaric acid, VC/maleic anhydride, VC/acrylic acid, VC/methacrylic acid, VC/acrylic or methacrylic ester. Satisfactory results were obtained with PVC in an amount 10-70 wt %, more preferably 20-50 wt %.

The EPDM, EPR or other polyolefinic elastomer rubbers employed in the present invention may be any of those known in the art, including grafted EPDMs and/or EPRs and/or other polyolefinic elastomers and mixtures thereof. Suitable grafted monomers would be, but are not restricted to,

maleic anhydride, acrylic acids, methacrylic acids, hydroxy methacrylates, silanes, allyl glycidyl ether or butendiol. The amount of EPDM and/or EPR employed was 15-70 wt %, more preferably 20-50 wt %.

The polycaprolactone or *in situ* polymerised  $\epsilon$ -caprolactone is a polyester with any type of end group. The amount of PCL should be in the range 5-50 wt %, more preferably 8-26 wt %.

The elastomeric properties (the compression set value) is greatly improved by the use of cross-linking agents. This could be any sulphur curing system suitable for steam or press curing, including but not restricted to i) mercaptobenzothiazole (MBT), tetramethylthiuram monosulphide and sulphur; ii) zinc oxide, stearic acid, tetramethylthiuram disulphide (TMTD), MBT and sulphur;

iii) MBT, TMTD, tellurium diethyldithiocarbamate (TEDC) and sulphur; iv) MBT, TMTD, zinc dibutyldithiocarbamate (ZBDC), TEDC and sulphur; v) ZBDC, tetraethylthiuram disulphide, zinc dimethyldithiocarbamate (ZMDC), 4,4'-dithiobis(morpholine) and sulphur; vi) TMTD, ZBDC, ZMDC, 4,4'-dithiobis(morpholine) and sulphur. It could also be a peroxy curing agent, referring to any conventional known peroxide and hydroperoxide with a reasonable half-life, including but not restricted to *tert*-butylperoxy-3,3,5-trimethylhexanoate, *tert*-butylperoxy isopropyl carbonate,  $\alpha,\alpha'$ -bis(*tert*-butylperoxy) diisopropylbenzene, *tert*-butyl peroxyacetate, dicumyl peroxide, 2,5-dimethyl-2,5-di-*tert*-butylperoxyhexane (i.e. 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane), 1,1-bis(*tert*-butylperoxy)-3,3,5-trimethylcyclohexane, *tert*-butyl cumyl peroxide, bis(*tert*-butyl peroxyisopropyl) benzene (i.e. bis[1-(*tert*-butylperoxy)-1-methylethyl]benzene), 2,5-dimethyl-2,5-di-*tert*-butylperoxy-3-hexyne (i.e. 2,5-bis(*tert*-butylperoxy)-2,5-dimethyl-3-hexyne), *n*-butyl-4,4-bis(*tert*-butylperoxy) valerate, *tert*-butyl peroxybenzoate and di-*tert*-butyl peroxide (i.e. *tert*-butylperoxide). It could also be an alternative chemical crosslinking agent, including but not restricted to poly(ethylene glycol), 1,6-hexanediol, 1,6-hexanediamine, methylene diphenyl-diisocyanate, 1,6-hexane diisocyanate, tolylene diisocyanate, di-, tri- or tetraglycidylethers, phthalic anhydride, trimellitic acid, iso- or orthophthalic acid. A combination of a chemical crosslinking agent and sulphur or peroxide curing agents could further improve the blends. The amount of crosslinking agents should be 0 to 10 wt%, preferably 0.05 to 10 wt%, especially 0.1-

10 wt % or more preferably 0.2-4.5 wt%.

In the present invention a crosslinking coagent may also be used, including but not restricted to diallyl phthalate, diebezoyl-*p*-quinone dioxime, triallyl cyanurate, ethylene dimethacrylate, pentaerythritol tri(meth)acrylate, trimethylolpropane tri(meth)acrylate, triallyl isocyanurate, pentaerythritol tetramethacrylate, diallyl maleate and *N,N'*-*m*-phenylenedimaleimide. The amount of coagent is from 0 to 30 wt %, preferably 0.05 to 30 wt% or, more preferably, from 0.4 to 10 wt %.

There is no particular restriction to the plasticizer used. Any plasticizer that is useful for the production of a plasticized vinyl chloride resin product may be used. It may be, but is not restricted to, monocarboxylic acid esters, acetates, propionates, butyrates, glycolic acid esters, benzoic acid esters, epoxidized acid esters, phosphates, trimellitic acid esters, polymeric plasticisers, phthalates, citric acid esters, sulphonic acid esters and sulphamides. The amount is 0-50 wt % , more preferably 8-23 wt %.

The stabilizers and processing aids used in this invention may be one or more of the commercially available compounds well known in the art. Preferred stabilizers include, but are not restricted to stabilizers based on lead, barium/zinc, calcium/zinc, organotin stabilizers, organotin mercaptides and -sulphides, metal carboxylate stabilizers, barium/cadmium, and metal free stabilizers. Co-stabilizers such as organic phosphites, epoxy compounds, polyols, antioxidants, 1,3-diketones, dihydropyridine and *b*-ketocarboxylic acid esters may also be used. Preferred processing aids include, but are not restricted to fatty alcohols and dicarboxylic acid esters, fatty acid esters of short-chain alcohols, (metal salts of) fatty acids and -amides, oligomeric fatty acid esters, fatty alcohol fatty esters, wax acids, including their esters and soaps, polar polyethylene waxes, nonpolar polyolefin waxes, paraffin waxes, fluoropolymers, and combinations thereof. The total amount of stabilizers and processing aids used in blends of the present invention is 0.3-6 wt %, more preferably 1.5-5 wt %.

## EXAMPLES

Tensile tests were performed according to ISO R527 standards. Compression set tests were run to ISO 1856 standards at 25% deflection. Congo red measurements were carried out at 200 °C in glass tubes (d = 3 mm, h = 3 cm material).

The present invention is illustrated by the following, non-limiting examples. The denomination for all the numbers given in the Tables is phr (parts per hundred). Five PVC masterbatches were employed:

PVC masterbatch	1	2	3	4	5
Norvinyl S 7065 <sup>1</sup>	100	100	100	100	
Norvinyl S 805 <sup>1</sup>					100
Diisodecylphthalate(DIDP) <sup>2</sup> 30		50	50		
Dioctylphthalate (DOP) <sup>2</sup>				50	
Diethylhexylphthalate (DEHP) <sup>2</sup>					54.6
Lancrostab 242 <sup>3</sup>	2	2			
Bärostab 777x <sup>4</sup>			2	2	
Stabilox CZE2040 <sup>5</sup>					4
Epoxidized soybean oil (ESO) <sup>6</sup> 3		3	3	3	3
Allied 316A <sup>7</sup>				0.4	
PVC mb, total phr	135	155	155	155	159

1: PVC, Hydro Polymers Nordic; 2: Plasticizer, Exxon; 3: Stabilizer, Acros; 4: Stabilizer, Bärlocher; 5: Stabilizer, Henkel; 6: Costabilizer, Henkel; 7: Lubricant, Allied Chemicals Ltd.

Example 1.

Blends with PCL/EPDM-ratio 1:1 were prepared with and without curatives and transesterification catalyst. The results are given in table 1.

Table 1.

Compound no.	1	2	3	4
PVC masterbatch 1	135	135	135	135
Royaltuf 465 <sup>1</sup>	50	50	50	50
Tone P787 <sup>2</sup>	0	55	55	55
Perkadox BC <sup>3</sup>	0	1	0	1
Prosper DBTL/C <sup>4</sup>	0	0	1	1
Compr.set (%)	85	54	58	47
Congo red (min.)	14.5	7.5	7	8
Tens.strength (MPa)	2.7	4.9	4.3	13.0
Max. elongation (%)	4	95	188	396

1: EPDMgMAH, Uniroyal; 2: PCL, Union Carbide; 3: Peroxide, Akzo; 4: Transesterification catalyst/stabilizer, Comtin s.r.l.

The elasticity (compression set value) clearly improves by introducing a peroxy curing agent or a transesterification catalyst. Combining both chemicals has the best effect. The tensile strength and elongation at break are clearly improved by employing PCL as a compatibilizer.



Example 2.

Blends with increased amount of EPDMgMAH (EPDM grafted with maleic acid anhydride) were prepared. The results are given in table 2.

Table 2.

Compound no.	5	6	7	8
PVC masterbatch 2	155	155	155	155
Royaltuf 465 <sup>1</sup>	100	100	155	300
Tone P787 <sup>2</sup>	0	30	45	45
Perkadox BC <sup>3</sup>	0	1	1.55	1.55
Sartomer 351 <sup>4</sup>	0	0	0	3.1
Prosper DBTL/C <sup>5</sup>	0	1	1.55	1.55
Compr.set (%)	72	47	43	33
Congo red (min.)	4	14.5	12	10
Tens.strength (MPa)	1.3	6.0	6.6	5.1
Max. elongation (%)	25	240	288	253

1:EPDMgMAH, Uniroyal; 2: PCL, Union Carbide; 3: Peroxide, Akzo; 4: Crosslinking coagent, Cray Valley Photocure; 5: Transesterification catalyst/stabilizer, Comtin s.r.l.

Increased amount of the rubber component has a positive effect on the elasticity, when a cross-linking agent is employed (cf. compounds no. 1 & 5 and 4 & 8). Introducing a crosslinking co-agent further enhances the elasticity.

A change in stabilizer to another Ba-Zn type, tin or lead stabilizer improved the thermal stability, but had a slightly detrimental effect on the compression set. The best result was achieved with a Ba-Zn stabilizer. The results are given in table 3.

Table 3.

Compound no.	9	10	11
PVC masterbatch 3	155	155	155
Royaltuf 465 <sup>1</sup>	300	300	300
Tone P787 <sup>2</sup>	0	45	45
Perkadox BC <sup>3</sup>	0	1.55	1.55
Sartomer 351 <sup>4</sup>	0	3.1	0
Sartomer 295 <sup>4</sup>	0	0	3.1
Prosper DBTL/C <sup>5</sup>	0	1.55	1.55
Compr.set (%)	82	38	43
Congo red (min.)	1.3	23	31
Tens.strength (MPa)	0.7	6.4	6.5
Max. elongation (%)	46	339	300

1:EPDMgMAH, Uniroyal; 2: PCL, Union Carbide; 3: Peroxide, Akzo; 4: Crosslinking coagent, Cray Valley Photocure; 5: Transesterification catalyst/stabilizer, Comtin s.r.l.

Example 3.

Properties of blends where EPDMgMAH is exchanged with unmodified EPDM are given in table 4.

Table 4.

Compound no.	12	13	14	15
PVC masterbatch 3	155	155	155	
PVC masterbatch 4				155
Nordel 2760 <sup>1</sup>	300	200	300	300
Tone P787 <sup>2</sup>	0	30	45	45
Perkadox BC <sup>3</sup>	0	1	1.55	1.55
Sartomer 351 <sup>4</sup>	0	3.1	0	0
Perkalink 300 <sup>5</sup>	0	0	3.1	3.1
Prosper DBTL/C <sup>6</sup>	0	1	1.55	1.55
Compr.set (%)	86	52	44	48
Congo red (min.)	6.0	18.5	47	51
Tens.strength (MPa)	0.1	6.5	4.1	2.3
Max. elongation (%)	27	151	234	113

1:EPDM, Du Pont; 2: PCL, Union Carbide; 3: Peroxide, Akzo; 4: Crosslinking coagent, Cray Valley Photocure;  
5: Crosslinking coagent, Akzo Nobel, 6: Transesterification catalyst/stabilizer, Comtin s.r.l.

The elasticity (compression set value) was slightly worsened (compound no. 10,11 : 14, 15). The thermal stability improved, but the tensile properties also decreased. EPDMgMAH therefore seems to be a better choice, even though plain EPDM also provide TPE's with good elasticity.

Example 4.

Properties of blends of PVC, PCL and EPRgMAH (EPR grafted with maleic acid anhydride) with increasing amount of an alternative chemical crosslinking agent are given in table 5.

Table 5.

Compound no.	16	17	18	19
PVC masterbatch 5	159	159	159	159
Exxelor 1801 <sup>1</sup>	108	108	108	108
Tone P787 <sup>2</sup>	54	54	54	108
Toluene diisocyanate (TDI) <sup>3</sup>	0	6.5	17.5	22
Prosper DBTL/C <sup>4</sup>	1	1	1	1
Compression set (%)	73	74	76	75
Congo red (min.)	67	27	30.5	40.5
Tens. strength (MPa)	12	15	38	33
Max. elongation (%)	378	385	510	790

1: EPRgMAH, Exxon; 2: PCL, Union Carbide; 3: Chemical crosslinking agent;

4: Transesterification catalyst/stabilizer, Comtin s.r.l.

The compression set is high and the morphology is poorer than that obtained with peroxy curing agents, but the tensile properties are superior to those obtained for similar blends.

Example 5.

Properties of blends of PVC, PCL and EPRgMAH with varying coagents are given in table 6.

Table 6.

Compound no.	20	21	22
PVC masterbatch 3	155	155	155
Exxelor 1803 <sup>1</sup>	300	300	300
Tone P787 <sup>2</sup>	0	45	45
Perkadox BC <sup>3</sup>	0	1.55	1.55
Sartomer 295 <sup>4</sup>	0	3.1	0
Sartomer 351 <sup>4</sup>	0	0	3.1
Prosper DBTL/C <sup>5</sup>	0	1.55	1.55
Compr.set (%)	55	47	44
Congo red (min.)	23	29	29
Tens.strength (MPa)	0.65	1.18	1.43
Max. elongation (%)	79	140	156

1: EPRgMAH, Exxon; 2: PCL, Union Carbide; 3: Peroxide, Akzo; 4: Coagent, Cray Valley Photocure;

5: Transesterification catalyst/stabilizer, Comtin s.r.l.

EPRgMAH provides TPE's with good elasticity and thermal stability, but decreased tensile properties compared with blends with EPDMgMAH as the rubber agent (cfr. compound no. 8, 10, 11 & 21, 22).

**Example 6.**

Two more masterbatches were employed.

Masterbatch	6	7
Vinolit VK801 <sup>1</sup>	100	100
Diisodecylphthalate (DIDP) <sup>2</sup>	50	
Bärostab 777x <sup>3</sup>	2	2
Epoxidized soybean oil (ESO) <sup>4</sup>	4	4

1: PVC-g-EVA, Vinolit; 2: Plasticizer, Exxon; 3: Stabilizer, Bärlocher; 4: Co-stabilizer, Henkel..

Properties of blends of PVCgEVA (PVC grafted with ethylene/vinyl acetate) and EPDMgMAH are given in table 7.

**Table 7.**

Compound no.	23	24	25	26
Masterbatch 6	100	100	100	
Masterbatch 7				100
Nordel 5892 <sup>5</sup>	200	200	200	200
Perkadox BC <sup>6</sup>		0.5	1	
Compr.set (%)	49	44	40	52
Congo red <sup>7</sup> (min.)	33	32	29.5	21
Tens.strength (MPa)	6.24	4.39	4.47	9.46
Max. elongation (%)	803	455	379	772

5: EPDM, Du Pont; 6: Peroxide, Akzo; 7: 180 °C.

By exchanging PVC with a copolymer (PVCgEVA) TPE's with excellent elasticity, thermal and tensile properties were obtained.

### CLAIMS

1. A thermoplastic elastomeric composition comprising PVC, homo- or copolymer, EPDM and/or EPR and/or other polyolefinic elastomer, polycaprolactone (PCL) and/or *in situ* polymerised  $\epsilon$ -caprolactone, and conventionally known additives for the preparation of PVC.
2. A thermoplastic elastomeric composition comprising 10-70 weight % PVC, homo- or copolymer, 15-70 weight % EPDM and/or EPR and/or other polyolefinic elastomer, 5-50 weight % polycaprolactone (PCL) and/or *in situ* polymerised  $\epsilon$ -caprolactone, 0-50 weight % plasticizer, 0.3-6 weight % stabilizer and other conventionally known additives for the preparation of PVC.
3. A thermoplastic elastomeric composition according to claim 1 or claim 2, wherein the EPDM and/or EPR and/or other polyolefinic elastomer are modified with one or more grafted monomers selected from maleic anhydride, acrylic acid, methacrylic acid, hydroxy methacrylates, silanes or other suitable monomers.
4. A thermoplastic elastomeric composition according to any one of the preceding claims, wherein the composition comprises 0-10 weight % sulphur-, peroxy curing or chemical crosslinking agent.
5. A thermoplastic elastomeric composition according to claim 4, wherein the composition comprises 0-30 weight % coagent for the crosslinking agent.
6. A thermoplastic elastomeric composition according to any one of the preceding



## CLAIMS

1. A thermoplastic elastomeric composition comprising PVC, homo- or copolymer, EPDM and/or EPR and/or other polyolefinic elastomer, polycaprolactone (PCL) and/or *in situ* polymerised  $\epsilon$ -caprolactone, and conventionally known additives for the preparation of PVC.
2. A thermoplastic elastomeric composition comprising 10-70 weight % PVC, homo- or copolymer, 15-70 weight % EPDM and/or EPR and/or other polyolefinic elastomer, 5-50 weight % polycaprolactone (PCL) and/or *in situ* polymerised  $\epsilon$ -caprolactone, 0-50 weight % plasticizer, 0.3-6 weight % stabilizer and other conventionally known additives for the preparation of PVC.
3. A thermoplastic elastomeric composition according to claim 1 or claim 2, wherein the EPDM and/or EPR and/or other polyolefinic elastomer are modified with one or more grafted monomers selected from maleic anhydride, acrylic acid, methacrylic acid, hydroxy methacrylates, silanes or other suitable monomers.
4. A thermoplastic elastomeric composition according to any one of the preceding claims, wherein the composition comprises 0-10 weight % sulphur-, peroxy curing or chemical crosslinking agent.
5. A thermoplastic elastomeric composition according to claim 4, wherein the composition comprises 0-30 weight % coagent for the crosslinking agent.
6. A thermoplastic elastomeric composition according to any one of the preceding

claims, wherein the composition comprises 20-50 weight % EPDM and/or EPR and/or other polyolefinic elastomers modified with grafted maleic anhydride, acrylic acid, methacrylic acid, hydroxy methacrylates, silanes or other suitable monomers.

7. A thermoplastic elastomeric composition according to any one of the preceding claims, wherein the content of PCL is 5-30 weight %.
8. Use of a thermoplastic elastomeric composition according to any one of the preceding claims for uPVC window and other frame material seals, vehicle window seals and waistrails, technical and appliance hose, automotive components, speciality electrical cabling (appliance and data), O-rings, gaskets or seals.



Application No: GB 9927333.6  
Claims searched: 1-8

Examiner: Jason Scott  
Date of search: 13 March 2000

**Patents Act 1977**  
**Search Report under Section 17**

**Databases searched:**

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK CI (Ed.R): C3M

Int CI (Ed.7): C08L

Other: ONLINE: WPI, JAPIO, EPODOC

**Documents considered to be relevant:**

Category	Identity of document and relevant passage	Relevant to claims
X	US 4442253 EXXON See whole document and in particular column 7, lines 49-55, example 1 and 2-1, 2-5, 2-6 in table II.	1-4, 6 & 7
X	US 4421882 EXXON See whole document and in particular column 7, lines 42-48, example 1 and 2-1, 2-6 in table II.	1-4, 6 & 7
X	WPI Abstract Accession No 1986-314242 & JP 61231051 A SUMITOMO ELECTRIC (15.10.86) See abstract	1 & 3

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.